

MANNED SPACECRAFT CENTER

Internal Note No. MSC-IN-65-EP-10

MEASUREMENT OF THE MELTING AND EVAPORATION RATE OF FROZEN
PARTICLES OF NITROGEN TETROXIDE AND MONOMETHYLHYDRAZINE

Prepared by:

Herschel H. Jamison
Herschel H. Jamison
Chief Chemist, Thermochemical Test Branch
Materials Laboratory

Approved by:

Jesse C. Jones
Jesse C. Jones
Chief, Thermochemical Test Branch

Approved by:

Joseph G. Thibodaux, Jr.
Joseph G. Thibodaux, Jr.
Chief, Propulsion and Power Division

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INTRODUCTION

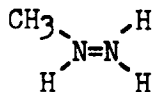
This test program was initiated for the purpose of determining the time required for frozen particles of propellants to melt and evaporate under Gemini spacecraft conditions. The test was accomplished in order to support the Gemini GT-4 mission during which the egress and ingress hatch was opened and the hazards associated with introduction of propellants into the cabin existed. Various size droplets of monomethylhydrazine and nitrogen tetroxide were frozen and subsequently placed in a flask containing oxygen at ambient temperature and at pressures of 5, 3, 2, 1, and 0.5 psia. The tests were accomplished in a dry box with a nitrogen dry atmosphere.

The test program was performed by the Thermochemical Test Branch Materials Laboratory in coordination with the Auxiliary Propulsion and Pyrotechnics Branch of the Propulsion and Power Division. The test was accomplished in the Materials Laboratory, Building 350, Site 1, during the period from May 10, 1965 to May 14, 1965.

DESCRIPTION OF TEST MATERIALS

Monomethylhydrazine

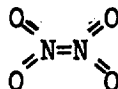
Monomethylhydrazine is a rocket engine fuel procured in accordance with specification MIL-P-27404. Its chemical formula is as follows:



Monomethylhydrazine is an extremely toxic material and the physiological hazards associated with exposure to its vapors are significant.

Nitrogen Tetroxide

Nitrogen tetroxide is a rocket engine oxidizer procured in accordance with specification MIL-D-26539 (USAF). The chemical formula is as follows:



It, likewise, is a very toxic material and exposure to its vapors is hazardous to human beings as well as deleterious to various materials such as space suit materials.

DESCRIPTION OF TEST APPARATUS

The experiments on frozen hypergolic propellants were performed within a one (1) liter round bottom boiling flask with an outer standard taper neck (Figures 1 and 2). The flask was modified to incorporate a thermocouple inserted through a septum and to provide an inlet and exhaust connection for vacuum and oxygen. An aluminum plate was provided for attaching the rods supporting the flask and the liquid nitrogen dewar; and for mounting the guide for the glass filament and stopper. The glass filament and stopper was constructed from an inner standard taper ground glass joint with a tube provided on either end. The lower tube was drawn out to a filament of about 300 microns in diameter with a bead on the end of about 350 microns in diameter (see Figure 3).

This apparatus was placed inside a dry box and connections were made for oxygen supply, vacuum, pressure readout and dry box exhaust. In addition to the test apparatus, other materials and equipment within the dry box consisted of a dewar containing liquid nitrogen for replenishing the main liquid nitrogen dewar and a dewar with crushed ice to keep the propellants chilled below their vaporizing points. The interior of the dry box was flushed with gaseous nitrogen to provide a moisture free and inert environment.

Outside the dry box, connections to the vacuum pump, manometer, and oxygen cylinder were made through appropriate valves to facilitate making the proper changes of the atmosphere in the test flask. A potentiometer was used to read the temperature within the flask.

TEST PROCEDURE

A droplet of the test material (see Figure 3) was transferred to the glass filament and lowered into the LN_2 vapor where it solidified. A measurement was then made visually by holding a metric scale, graduated in one half millimeter units, behind the frozen droplet (see Figure 4). The frozen droplet was then inserted into the oxygen atmosphere of the flask with the assistance of the guide rods. The stop watch was then started and the flask evacuated to the desired absolute pressure for the particular test run (see Figures 5 and 6). The time, in seconds, when the droplet returned to the liquid state, and at the completion of evaporation was recorded. The flask was then flushed with oxygen and prepared for the next test run. Temperature readings were taken at the beginning, during the test run, and at the end of the test run prior to flushing the flask with oxygen.

Oxidizer and fuel test runs were accomplished under as near identical conditions as was possible. The initial frozen particle temperature approximated that of liquid nitrogen ($-160^\circ\text{C}.$). No variation in initial temperature was attempted.

It was not possible to positively control the size of the particles and the accuracy of the measurement of the frozen particle varied some 250 microns. Also, there was some difficulty in obtaining a completely inert atmosphere in the dry box; consequently, the samples did not remain moisture free and fresh samples were required for each test run. The monomethylhydrazine samples inside the dry box were maintained at $0^\circ\text{C}.$ with ice, and, for the N_2O_4 , salt was added to the ice water mixture to lower the temperature to approximately $-10^\circ\text{C}.$

RESULTS AND DISCUSSION

The monomethylhydrazine (MMH) stored in the dry box and the eye dropper used for transferring fuel droplets to the glass filament stopper were maintained at 0°C. This was accomplished by storing the fuel in the eye dropper, which in turn was kept in a beaker immersed in an ice water bath. Contamination with moisture was avoided by replacing the quantity of fuel in the eye dropper frequently. Moisture contamination was easily determined in the case of monomethylhydrazine since the time for complete vaporization increased considerably when excessive moisture was present.

Uniform particles of monomethylhydrazine were readily formed and measurement of the frozen particle was quickly obtained. No problem was experienced in obtaining the required pressure in a short period of time since the vacuum pump was able to obtain low pressure rapidly.

The temperature inside the test flask at the beginning and end of each test run was maintained at ambient (approximately 23°C.). When the frozen particle was introduced into the flask, the temperature dropped to approximately 21.5°C. and then gradually returned to ambient temperature. Observation of the sample in the flask during evaporation was made difficult by the optics of the flask, especially at the end of the monomethylhydrazine (MMH) evaporation. It was determined that particles of monomethylhydrazine (MMH) having diameters ranging between 1000 and 2000 microns required from 5 to 10 minutes to evaporate. The results of the tests with monomethylhydrazine are tabulated in Table 1.

The viscosity, surface tension and ease of vaporization of nitrogen tetroxide is such that it was not possible to get a frozen particle of uniform size on the glass filament and measurement of the particle size was very difficult. In spite of these difficulties, however, adequate control was maintained so that sufficiently reliable information was obtained.

Another problem associated with the nitrogen tetroxide test phase involved the vacuum pump which was unable to pull down to the required pressure instantaneously, even though it was connected to a ballast tank that was evacuated. This was a serious problem during the 0.5 psia test run only, however. The length of time required to achieve the 0.5 psia was sufficient for the particle to vaporize before the required pressure was obtained. Thus, the results for this pressure range were somewhat erratic and subject to question.

For the tests involving nitrogen tetroxide, an ice bath with salt added was used to store the bulk nitrogen tetroxide in the dry box and the eye dropper used to transfer oxidizer droplets to the glass filament stopper was maintained at low temperature by placing it in

the liquid nitrogen vapors. Nitrogen tetroxide will rapidly absorb moisture from the atmosphere, therefore, it was necessary to change the oxidizer frequently to avoid this problem. As a result of this phase of the program, it was determined that particles of nitrogen tetroxide having diameters ranging from 1000 to 3000 microns required from 1 to 5 minutes to evaporate. The results of the tests with nitrogen tetroxide are tabulated in Table 2.

There appears to be no insurmountable problem in eliminating a propellant contaminated atmosphere within a spacecraft. The particles of frozen propellant will evaporate readily under Gemini spacecraft cabin pressure conditions and by subsequently exhausting and repressurizing, the contamination should ultimately be eliminated.

CONCLUSIONS

1. Small, frozen particles of monomethylhydrazine from 1000 to 2000 microns in size will vaporize in 5 to 10 minutes under Gemini spacecraft cabin conditions.
2. Frozen particles of nitrogen tetroxide from 1000 to 3000 microns in diameter will vaporize in 1 to 5 minutes under Gemini spacecraft cabin conditions.
3. It appears that propellant vapors and/or particles, which may be inadvertently introduced into the cabin of the Gemini spacecraft, may be eliminated by some method such as alternately exhausting and repressurizing the cabin.

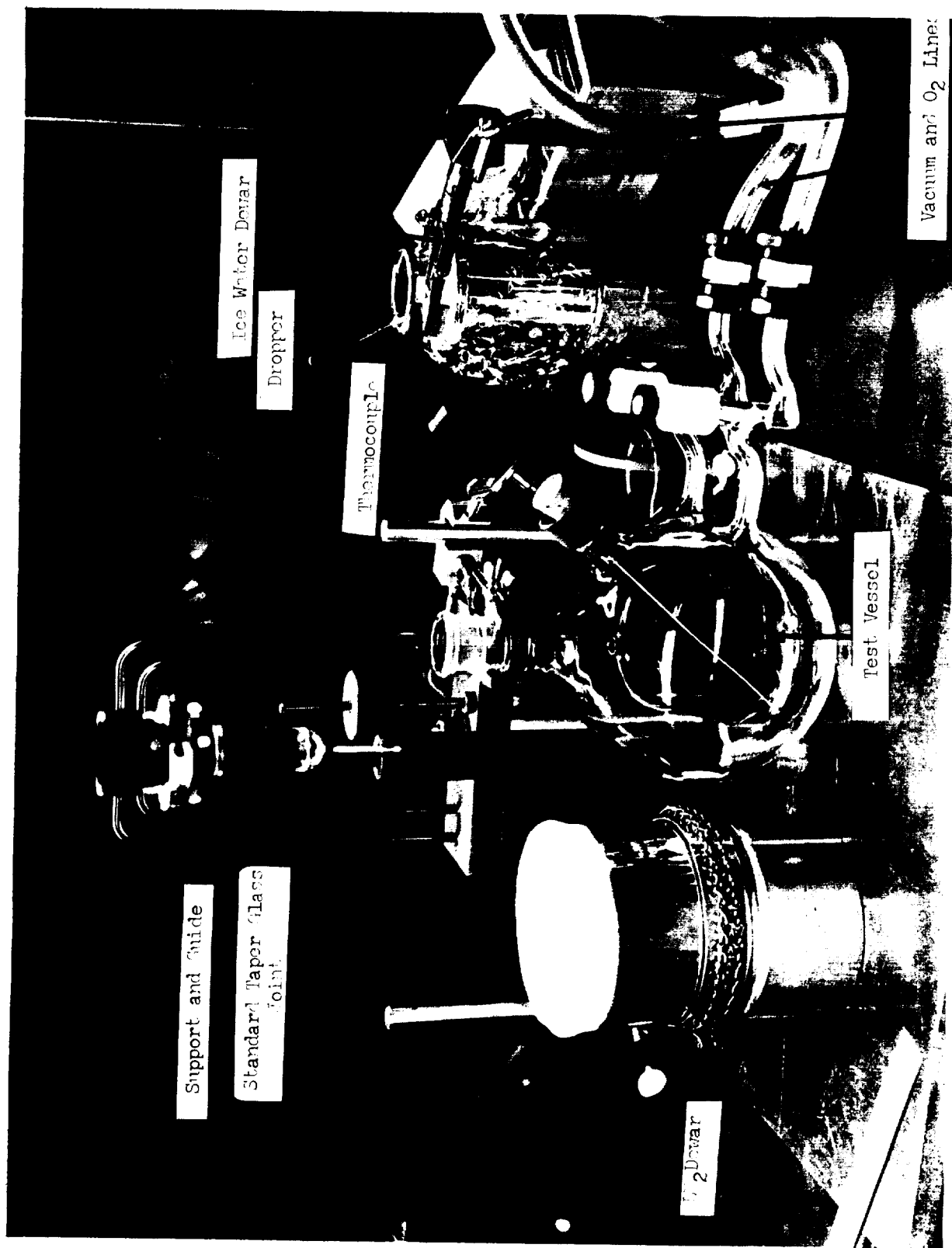


FIG. 2

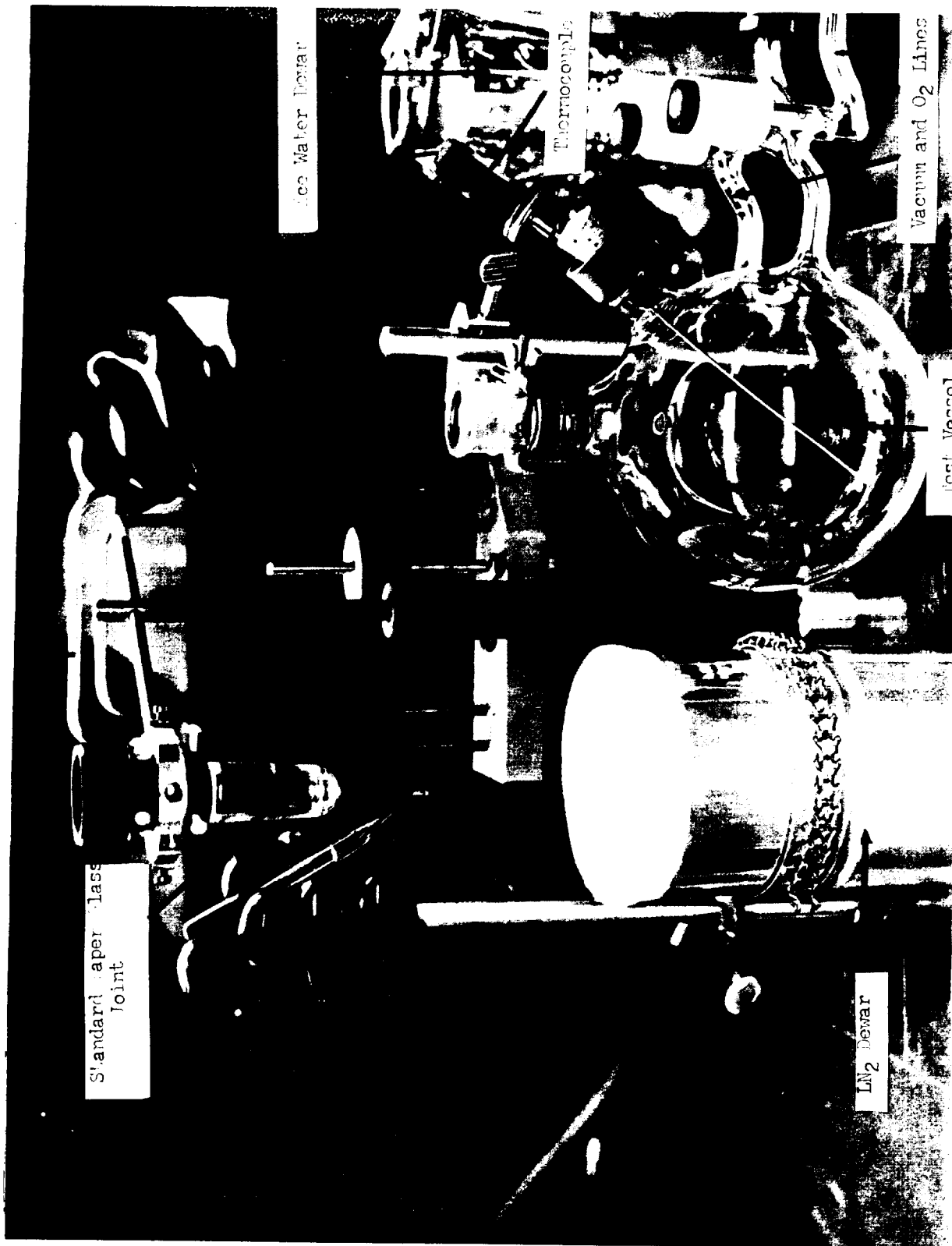


FIG. 3



FIG. 4

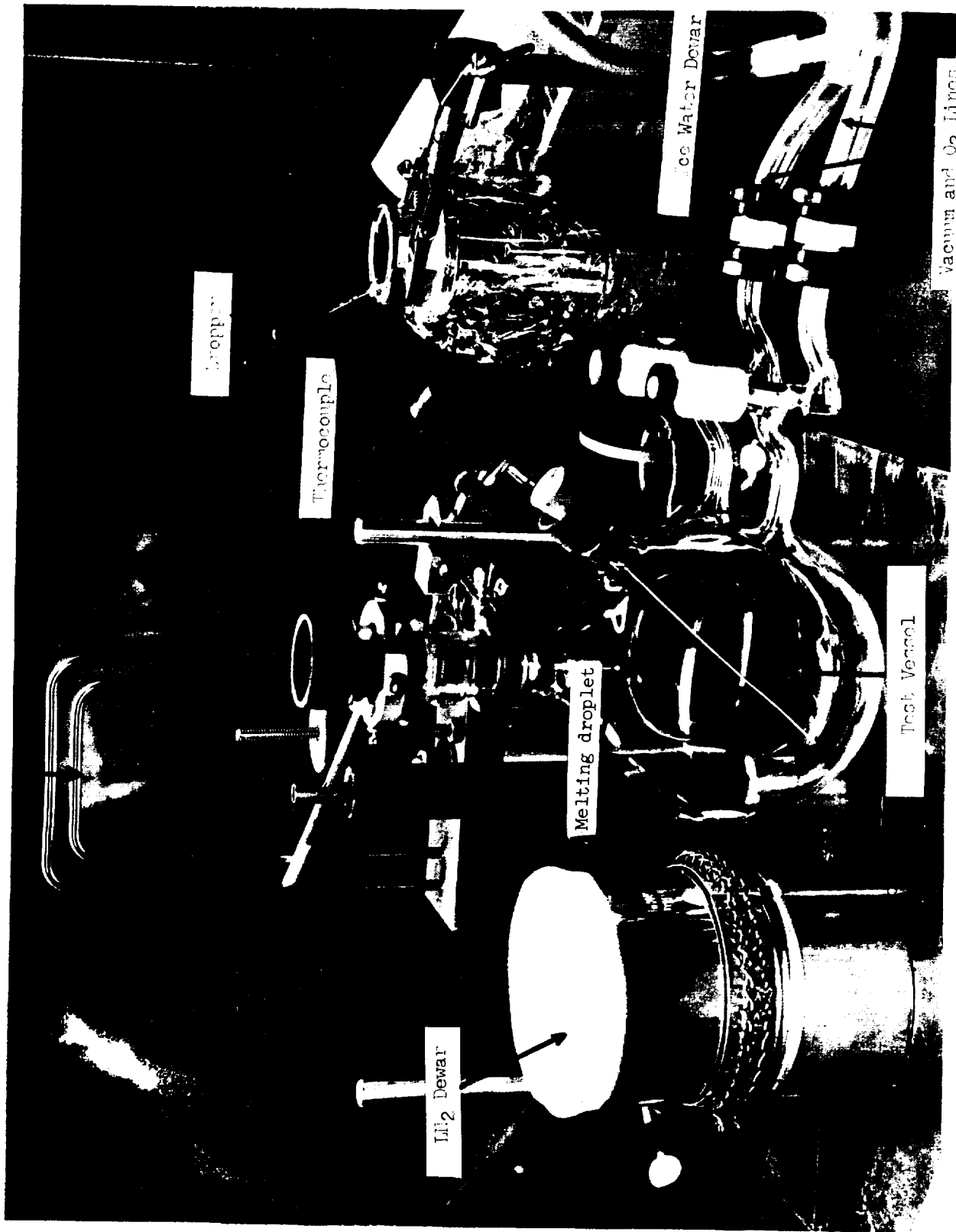


FIG. 5

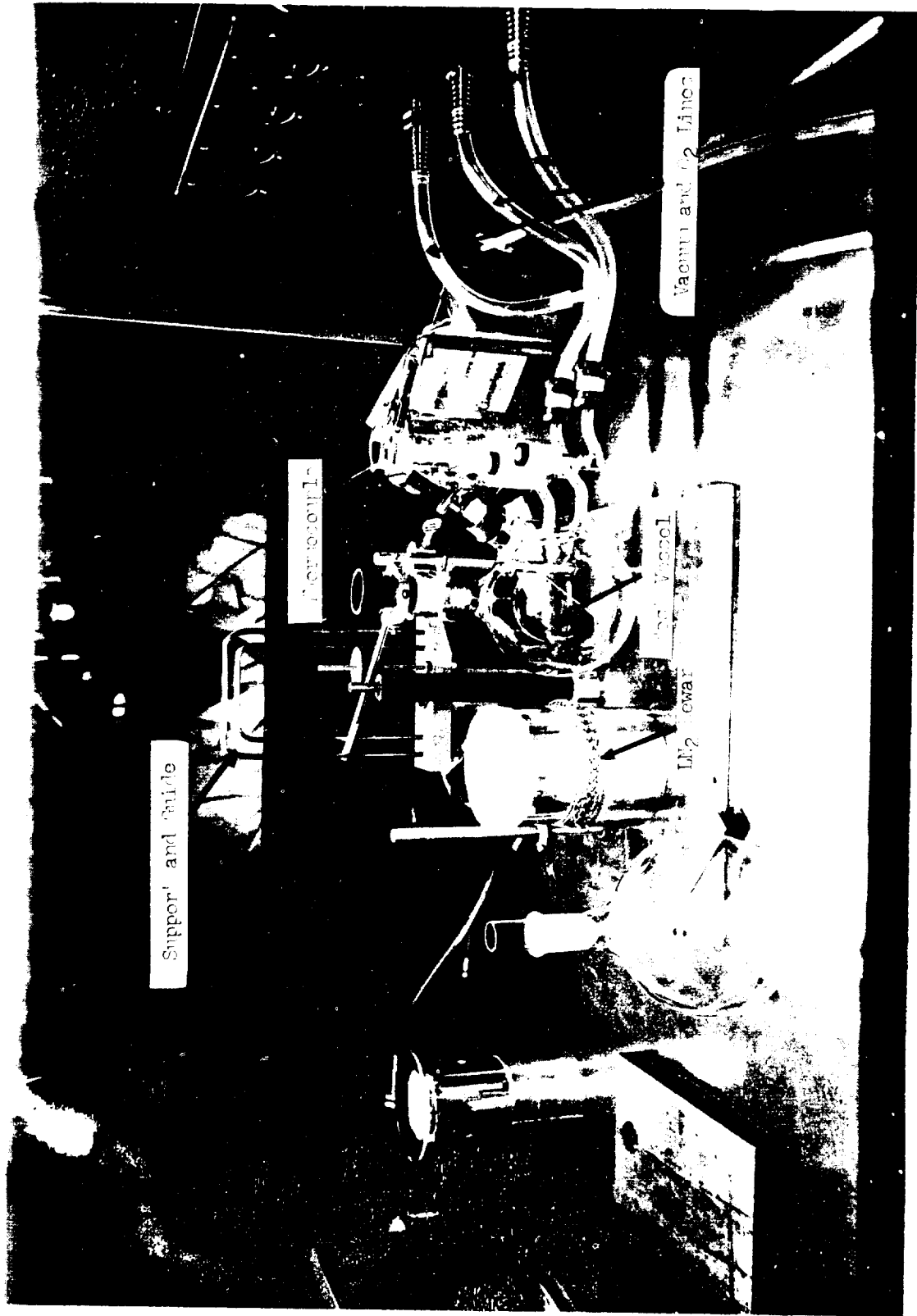


FIG. 6

TABLE 1

Monomethylhydrazine Data

Pressure (PSIA)	Run No.	Temperature, Degrees, C.	Pressure (Millimeters Hg.)	Particle size, Microns	Evaporation Time, Min:Sec.
5	1	23	508	1400	7:00
	2	23	508	1700	9:00
	3	23	508	1650	8:30
	4	23	508	1400	7:00
3	1	24	608	1200	5:00
	2	23	608	1300	5:00
	3	23	608	1600	7:00
	4	24	608	1100	4:30
2	1	22	658	1600	7:00
	2	22	658	1600	9:00
	3	23	658	1400	5:00
	4	23	658	1400	6:30
1	1	24	710	1900	8:00
	2	24	710	1100	4:00
	3	23	710	1100	4:00
	4	24	710	1100	4:30
0.5	1	22	734	1300	4:00
	2	23	734	1600	4:30
	3	22	734	1100	2:30
	4	22	734	1600	4:30

TABLE 2

Nitrogen Tetroxide Data

Pressure (PSIA)	Run No.	Temperature, Degrees, C.	Pressure (Millimeters Hg.)	Particle size, Microns	Evaporation Time, Min:Sec.
5	1	22	508	1600	3:00
	2	23	508	1200	2:30
	3	23	508	1000	2:30
	4	22	508	2500	4:30
3	1	24	608	1000	2:00
	2	23	608	3000	4:30
	3	23	608	2000	3:15
	4	23	608	1300	2:00
2	1	24	658	750	1:15
	2	23	608	3000	4:30
	3	23	608	2000	3:15
	4	23	608	1300	2:00
1	1	23	710	2500	2:45
	2	24	710	2300	2:30
	3	24	710	2100	2:15
	4	23	710	1800	2:15
0.5	1	22	718	2000	2:15
	2	23	722	1300	1:30
	3	23	718	2200	2:30
	4	24	726	3000	3:00